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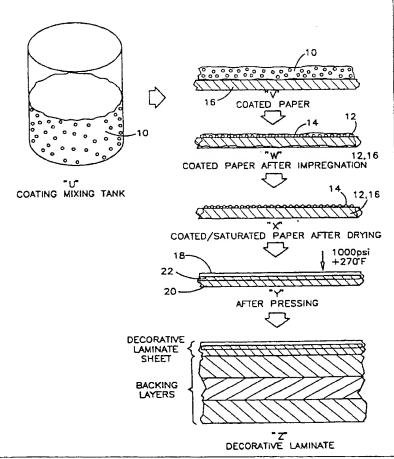
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(54) Title: DECORATIVE SURFACE LAYER AND PROCESS FOR ITS PRODUCTION

(57) Abstract

A decorative laminate (z) surface layer composition is prepared by selectively applying dissimilar thermoset or thermoplastic polymers (10) to a decorative laminate facing sheet (y) to achieve a brillant visual or pearlescent appearance. The laminate also exhibits enhanced wearability, chemical, thermal or ultra-violet radiation resistance or abrasion resistance.



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#### DECORATIVE SURFACE LAYER AND PROCESS FOR ITS PRODUCTION

#### FIELD OF THE INVENTION

The present invention relates to processes for achieving decorative laminates having a surface coating of dissimilar laminate resins. The laminates are suitable for counter tops, wall panels, floor surfacing, tabletops and the like.

### 10 <u>BACKGROUND</u>

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Decorative laminates have been conventionally made by stacking a plurality of layers of paper impregnated with thermosetting resins. Conventional laminates are made of three essential layers: a core 15 layer, a decorative layer, and surface layer. core or backing layer constitutes a bottom or supporting layer onto which the other layers are bonded. In high pressure laminates, the core layer consists of a plurality of core sheets (for example, three to eight) made from phenolic resin impregnated 20 cellulosic sheets such as kraft paper. The core layers lies a decor sheet impregnated with melamine resin or some other desired impregnating resin such as phenolic, amino, epoxy, polyester, silicone, acrylic and diallyl phthalate resins to name but a 25 In low pressure laminates the core layer is more often a sheet of particle board, normally in the range of 3/8 inch to 1 inch thick. possible for the core layer for either high or low 30 pressure laminates to made from materials other than paper or particle board, such as cloth (e.g. linen or canvas), wood or mat materials.

The type of decor sheet or decorative facing is dictated by the ultimate product and can be a paper, cardboard, fabric (either woven or felt), or any

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fibrous or cellulosic fiber decorative sheet, such as viscose rayon fiber or wood pulp fibers of high alpha cellulose content, or other decorative material that would provide a desired aesthetic appearance which are well known in the art.

An overlay sheet is provided on top of the decor sheet which, in the laminate, is essentially transparent and provides protection for the decor sheet.

Improvements of this process are disclosed in Scher et. al. U.S. Patent Nos. 4,255,480; 4,263,081; 4,327,141; 4,395,452; 4,400,423; Re. No. 32,152; Ungar et. al. U.S. Patent No. 4,713,138; and O'Dell et al. U.S. Patent No. 4,567,087. These patents are commonly assigned herewith and their disclosures are incorporated by reference herein.

Scher et. al. Re. 32,152 teaches that compositions containing small mineral particles, which when coated without resin over unimpregnated printed paper, provide surprising and unexpected properties permitting such paper to be used in the preparation of decorative laminates without an overlay sheet. The resultant laminates are highly abrasion resistant.

This Scher coating composition is composed of a mixture of small particles of alumina or other abrasion resistant particles of average 20-50 micron particle size, and a lesser amount of micro-crystalline cellulose particles, both

dispersed in a stable, aqueous slurry. The particles of alumina, of small size such that they do not interfere with the visual effects in the final product, serve as the abrasion resistant material and the micro-crystalline cellulose

particles serve as the preferred temporary binder.

Scher further teaches that the binder must be compatible with the resin system later utilized in the laminating procedure, usually melamine resin or in the case of certain low-pressure laminates a polyester resin system, and the micro-crystalline cellulose serves this function as well as stabilizing the small particles of alumina of the surface of the print sheet.

Ungar et. al. U.S. Patent No. 4,713,138 teaches the process of depositing onto the surface of a 10 decor sheet an ultra-thin layer of abrasion resistant material, which material is substantially disclosed in U.S. Patent No. 4,255,480, simultaneously with the complete resin saturation of 15 the decor sheet in a single step operation. resin composition of the Ungar process acts as the carrier for the abrasion resistant material. abrasion resistant composition consists essentially of an abrasion resistant hard mineral of fine particle size, preferably about 20-50 microns, in 20 quantities sufficient to provide an abrasion resistant layer without interfering with visibility. The abrasion resistant mineral in Ungar is preferably alumina, silica or a mixture thereof. 25 Ungar further teaches the use of a binder material for such mineral. The binder material in Ungar is present in an amount sufficient to bind the abrasion resistant mineral to the surface of the decor sheet. Such binder material is preferably a mixture of 30 micro-crystalline cellulose with a minor amount carboxy methyl cellulose.

One such binder sold by FMC Corporation under the trademark "AVICEL" is a mixture of approximately 89% micro-crystalline cellulose and 11% carboxy 35 methyl cellulose. The abrasion resistant

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composition suitably contains 1-8 parts by weight of "Avicel" to 4-32 parts by weight of mineral particles preferably at a ratio of mineral particles to binder material of 4:1 to 1:2, and a quantity of 1 part of "AVICEL" per 2 parts of mineral particles has been found to be particularly suitable.

Ungar et. al. also teaches that small additional quantities of carboxy methyl cellulose and a small quantity of silane may be added to the composition. Also, it is preferable to include a small quantity of surfactant, as disclosed in U.S. Patent No. 4,255,480, and a small quantity of solid lubricant to provide scuff resistance, as disclosed in U.S. Patent No. 4,567,087 in those compositions.

Accordingly, the above discussed patents provide single and two stage processes for providing a thin or ultra thin abrasion resistant laminate surface applied to decor sheets. However, it has been a continuing problem in the industry to provide a chemical, stain and abrasion resistant laminate surface on a decor sheet suitable for horizontal surfaces having certain brilliant visual appearance such as a pearlescent effect.

While considerable activity in the field has

led to many decorative surface appearances, these
activities resulted in the development of processes
and compositions wherein the resin material was
impregnated into the structure of the paper and the
thin or ultra-thin layers of the laminate resin on

the surface. The prior processes have failed to
achieve laminate which meet all the international
standards for horizontal laminate surfaces while
retaining brilliant visual effects and none have
achieved a laminate having a pearlescent finish that
is suitable for horizontal surfaces.

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and luster.

### SUMMARY OF THE INVENTION

It is an object of this invention to provide products and methods for producing products which overcome the above mentioned problems encountered in this field.

It is a particular object to provide a laminate surface layer composition including a two layer coating of at least two dissimilar resin polymers to achieve desirable wearability, and chemical, thermal, resistance to ultra-violet radiation, as well as resistance to abrasion, while achieving a brilliant visual decorative appearance of the laminate surface layer. This brilliant visual appearance is remarkable for its rich depth of color

A further object of the present invention is to obtain a true pearlescent appearance in a laminate. The results of this invention are very surprising as the resins used in this invention have long been known in the laminates field. In addition to providing these products, it is yet another object of this invention to provide processes for achieving these laminates.

These and other objects of the invention are
achieved by applying a surface coating of a liquid
or particulate resin onto a conventional decorative
facing sheet (including, prints, solids, foils,
etc.) made from any type of desirable material such
as paper, fabrics, wood or other cellulosic

material. The surface coating resin may be applied
as a liquid dispersion of multiple dissimilar
polymers, such as a colloid, a mixture of polymer
particles suspended in a liquid resin, an emulsion,
or an aqueous dispersion of polymer particles in
water. Exemplary of suitable polymer particles for

use herein are polyester, polyurethane, polyvinyl chloride, epoxy, and acrylic, or mixtures thereof. For purposes of this invention the term "particles" or "particulates" is not limited to those materials which are solid at room temperatures.

BRIEF DESCRIPTION OF THE DRAWINGS

Figure 1 is a flow chart showing a one step method to achieve the present invention using schematic sectional views of the decorative paper and laminate in accordance with the present invention.

Figure 2 is a flow chart showing a two step method to achieve the present invention using schematic sectional views of the decorative paper and laminate in accordance with the present invention.

Figure 3 is a flow chart showing the transfer paper method to achieve the present invention.

Figure 4 is a flow chart illustrating a dry 20 powder deposit method of achieving the present invention.

Figure 5 is a flow chart illustrating a twosided coating method of achieving the present invention and obtaining an anticurl backing on the decorative sheet.

### DETAILED DESCRIPTION OF EMBODIMENTS

With reference to Fig. 1, a one step process is seen. The coating mix tank (U) contains a

30 dispersion of at least two dissimilar resins (10) -- an impregnating resin (12) and a coating resin (14), which will melt and flow under heat and pressure. Coating resin (14) can be a solid particulate or liquid globules insoluble in and dispersed within impregnating resin (12). The dispersion (10) is

then coated onto the decorative facing sheet (16) as illustrated by coated sheet (V). Impregnating resin (12) soaks into and impregnates the facing sheet (16) which causes the coating resin (14) to be 5 filtered out onto the exterior surface of the facing sheet (16). The coated sheet after impregnation (W) is than dried in the usual manner resulting in coated paper (X). Dried coated sheet (X) which has become impregnated with impregnating resin (12) has a surface coating of coating resin (14). The dried coated and impregnated sheet (X) is than subjected to the usual laminating conditions to form the decorative laminate sheet (Y) which has substantially two surface layers. These two resin 15 layers include a surface layer (18) consisting essentially of coating resin (14) and a second layer (20) consisting of impregnating resin (12) which is contained almost entirely within the sheet. There is a small interface portion (22) within the sheet 20 with contains both resins (12) and (14). decorative laminate sheet (Y) is then laminated under heat and pressure to the backing layer to produce the decorative laminate (Z).

a resin that permeates into the decorative facing sheet material and, when the appropriate backing layer is used, into the backing layer as well. The backing layer for this invention can be any of a number of supporting substrate material, including layered kraft paper, cardboard, particle board, fabric (woven, non-woven and felts), mat materials, wood products or other supporting substrate materials as would be dictated by the ultimate use of the final product. The decorative facing sheet suitable for this invention can be one of any number

of materials, including paper, foils, fabrics (woven, non-woven and felt materials) or wood products and would depend on the ultimate aesthetic and performance requirements for the finished product.

With reference to Fig. 2, the two step process is seen. The coating mixing tank (L) contains a dispersion (5) of an aqueous mixture and coating resin (14), which will melt and flow under heat and pressure. Coating resin (14) can be a solid 10 particulate or liquid globules insoluble in and dispersed within the aqueous mixture. dispersion (5) is then coated onto the decorative facing sheet (16) as illustrated by coated sheet 15 The facing sheet (16) is then dried in the usual manner to produce dried coated sheet (N). Dried coated sheet (N) is then coated, saturated and impregnated with impregnating resin (12) to form saturated sheet (0) where upon the impregnated facing sheet is then subjected to normal laminating 20 conditions to produce the decorate laminate sheet (P) which has substantially two surface layers. These two resin layers include a surface layer (18) consisting essentially of coating resin (14) which 25 has substantially displaced impregnating resin (12) on the surface. A second layer (20) consists of impregnating resin (12) which is contained almost entirely within the sheet. There is a small interface portion (22) within the sheet with contains both resins (12) and (14). The decorative laminate sheet (P) is then laminated under heat and pressure to the backing layer to produce the decorative laminate (Q).

In Figure 3 the transfer sheet process is seen. In this process an aqueous solution containing the surface coating resin particles and a binder (30) is spread onto one side of the transfer or release paper (32) and dried. The coated transfer paper (40) is then placed over the surface of a resin impregnated decorative facing sheet (34), which is on top of the supporting substrate or backing layer The throw away portion (42) of the transfer paper (32) is removed and the layered remaining 10 materials can be used to form a laminate (38). This is usually done as in a high pressure laminating process (about 800 to 1500 psi) or a low pressure lamination process which is typically used when the 15 supporting substrate is a particle fiber board or wood substrate. The temperature will vary depending on the resins used and would be readily known by one skilled in this art.

Figure 4 illustrates another method of

20 achieving the present invention. Figure 4 shows how
the surface coating resin particles (50) are
sprinkled via shaker tray (46) over the wet
impregnating resin formulation coated on the
decorative facing sheet (52). The wet resin

25 decorative facing sheet is being transported along a
conveyor system (44) into an oven (48), wherein the

surface coating resin particles are secured onto the surface of the facing sheet by drying the wet resin. The decorative facing sheet is then ready to be used on any type of desirable support substrate or backing layer to form a laminate in the conventional way.

Figure 5 illustrates a method of achieving the present invention that also achieves a decorative facing sheet that will not curl during handling. Figure 5 a first slurry mixture (61) containing the 10 surfacing coating resin particles is applied on a first surface of the decorative facing sheet (62) and another slurry mixture containing an impregnating resin (63), that may have the same composition as the first slurry mixture or may have 15 a different composition, is applied to a second surface of the decorative facing sheet (62). first coating (61) can be melamine, the coating described in U.S. Patent Re. No. 32,152 or can be 20 the coating having at least two dissimilar resins wherein the one resin melts and flows under heat and pressure as disclosed herein. The resin coatings are permitted to dry or are dried on the facing sheet (64) in an oven where it is then ready for use 25 in conventional high or low pressure laminating to

make a laminate (66) having a supporting substrate or backing layer.

All of the above described processes can be used in high and low pressure laminates and/or for use with transfer foils, wall covering (fabric, paper or non-woven backed), acrylic films, wood veneers, flooring materials and exterior siding materials.

### PREFERRED EMBODIMENTS

The product produced in accordance with this invention includes a decorative facing sheet laminated onto the exterior surface of a backing layer and a coating layer that is an integral part of the laminate on the exterior surface of the facing sheet to form an outer surface thereon.

polymer particulate resin that melt and flow under heat and pressure and which is dissimilar from the laminate impregnating resin. To achieve a pearlescent appearance, the exterior coating layer should have a refractive index in the finished cured laminate dissimilar from the refractive index of the pearlescent ink on the decorative facing sheet.

The coating layer is made from at least one

Such coating may optionally contain a mixture

of an abrasion resistant mineral and a stabilizing

suspending agent or binder material for said

mineral. The abrasion resistant mineral has a particle size of between 1-200 microns and is present in the mixture in a concentration sufficient to provide abrasion resistance without interfering with visibility.

In a preferred form, the coating layer of this invention includes a mixture of small particles of alumina or other abrasion resistant particles of between about 1-200 micron particle size, polymer 10 particulates of between sub-micron and 250 micron particle size and a lesser amount of micro-crystalline cellulose particles, all dispersed in a stable, aqueous slurry composition. To achieve a pearlescent appearance, the polymer particulates 15 have a refractive index in the finished cured laminate dissimilar to the refractive index of the pearlescent ink on the decorative facing sheet. When using the polymer particulate coating dispersion, the particulates are present in the 20 dispersion such that they melt and flow at the elevated temperatures and pressures of the laminating process.

The particles of alumina or other abrasion resistant particles are of a small size such that

they do not interfere with the visual effects in the final product and serve as the abrasion resistant

material. The micro-crystalline cellulose particles serve as the preferred temporary binder material or suspending agent. It will be understood that the binder material or suspending agent must be

5 compatible with the impregnating resin later utilized in the laminating procedure, usually melamine resin, or in the case of certain low-pressure laminates, a polyester resin. The micro-crystalline cellulose serves this function as

10 well as stabilizing the small particles of alumina of the surface of the print sheet.

The preferred coating layer composition
contains a mixture of small particles of alumina and
the polymer particulates and a lesser amount of

15 micro-crystalline cellulose particles, all dispersed
in water creating a slurry. There must be an amount
sufficient of the binder material or suspending
agent, such as a micro-cyrstalline cellulose, to
retain the mineral particles and polymer

20 particulates in place on the surface of the decor
facing sheet. The binding material should be able
to withstand the subsequent laminating conditions.
In general, it has been found that satisfactory
results are attained with about 5 to 10 parts by

25 weight of the micro-crystalline cellulose for about
20-120 parts by weight of the alumina and polymer

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particulate. However, it is possible to work outside this range. The quantity of water in the slurry is also dictated by practical considerations, since if there is too little water, the slurry becomes so thick that it is hard to apply. Similarly, if there is too much water, the slurry becomes so thin that it is difficult to maintain a consistent thickness during the coating operation due to running of the slurry. Thus, a slurry containing about 2.0 wt % micro-crystalline 10 cellulose and about 24 wt % alumina and polymer particulates, based on the amount of water, is stable, i.e., the alumina does not settle out; but if more than about 3.5 wt % micro-crystalline 15 cellulose and about 24 wt % alumina and polymer particulates, based on the amount of water, is used, the slurry becomes very thixotropic and difficult to apply.

The slurry composition also preferably contains
a small amount of wetting agent, preferably a
non-ionic wetting agent, and a silane. The quantity
of wetting agent is not critical, but only a very
small amount is desirable and excess quantities
provide no advantage and can cause disadvantages
during processing. The silane acts as a coupling
agent which chemically binds the alumina or other

inorganic particles to the melamine matrix after impregnation and curing. The use of silane provides better initial wear since the alumina particles are chemically bound to the melamine in addition to

5 being mechanically bound thereto and therefore stay in place longer under abrasive wear. The particular silane used should be selected from among the group making it compatible with the particular laminating resin used. (See the 1976-77 Edition of Modern

10 Plastics Encyclopedia, Page 160, which lists some silanes useful with melamine and polyester systems.)

Plastics Encyclopedia, Page 160, which lists some silanes useful with melamine and polyester systems.)

In this regard, silanes having an amino group, such as gamma-aminopropyltrimethoxy silane, are particularly effective for use with melamine resins.

and, in fact, as little as 0.5% based on the weight of the alumina is effective to enhance the abrasion resistance of the final laminate. A maximum quantity of about 2% by weight based on the weight of the alumina or other particles is suggested since greater quantities do not lead to any significantly better results and merely increase the cost of the raw materials. The decorative paper is then impregnated in the normal manner with a suitable

25 laminating resin, usually a thermosetting resin.

The polymer particulates can be selected from any of the traditional laminating resins. Enhanced wearability, chemical, thermal, resistance to ultra-violet radiation, and resistance to abrasion is possible by selecting the appropriate coating resin for a specific property. For instance, a vinyl-ester may be selected if a high resistance to mineral acids and mineral basis is desired. acrylic may be selected for ultra-violet radiation 10 stability. An epoxy may be selected if thermal resistance is desired and for a high chemical and stain resistance properties. In order to achieve the brilliant visual pearlescent effect, it is important to select a resin having a refractive index in the finished cured laminate dissimilar from 15 the refractive index of the pearlescent ink on the decorative facing sheet being used. The selection of polymer particulates is preferably made from the group consisting of polyester, polyurethane, epoxy, polyvinyl chloride and acrylic, or mixtures thereof. 20 In addition to alumina, abrasion resistant particles may be mineral particles such as silica, zicronum oxide, cerium oxide, glass beads and diamond dust or mixtures thereof.

25 Another preferred method for achieving the objects of this invention is by the process of

depositing on the surface of a decor sheet a dispersion of liquid dissimilar resins or layer of polymer particulates simultaneously with the complete resin saturation of the decor sheet in a single step operation, in which the resin may optionally act as a carrier for the abrasion resistant material.

This process by which the present invention is achieved is best described as follows:

- (a) preparing a coating dispersion of at least two dissimilar resins, wherein the first of said dissimilar resins is an impregnating resin and wherein the second of said dissimilar resin is the surface coating resin which melts and flows under heat and pressure, and a binder material that can retain the second dissimilar resin on the exterior facing surface of the decorative facing sheet and that is compatible with said impregnating resin and that will withstand subsequent laminating conditions;
  - (b) coating and impregnating an unsaturated decorative facing sheet in at least one step by coating said coating dispersion over the exterior facing surface of said sheet at a rate such that said unsaturated sheet becomes substantially saturated with said impregnating resin, and the

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second dissimilar resin is filtered onto said facing surface; and

(c) drying said coated and impregnated decorative sheet to obtain a decorative sheet suitable for pressing.

Optionally, a hard mineral of fine particle size in a concentration sufficient to provide abrasion resistant layer without interfering with visibility may be added to the coating mixture. hard mineral that may be used in the coating 10 composition is of fine particle size, preferably between about 1-200 microns, and used in quantities sufficient to provide an abrasion resistant layer without interfering with visibility. The hard mineral is preferably alumina, silica, zirconium oxide, cerium oxide, glass beads, and diamond dust or mixtures thereof. When using a hard mineral in the coating mixture, a binding material or suspending agent for such mineral may be necessary to retain the mineral particle on the exterior surface of the decorative facing sheet. The binder material or suspending agent should have the properties of being able to withstand the subsequent laminating conditions and wherein said binding material or suspending agent is compatible with the

impregnating resin. Such binding material or

suspending agent is used in an amount sufficient to bind the abrasion resistant mineral to the surface of the decor sheet.

The dissimilar resins may be either in liquid

or particulate form. The coating resin that must
melt and flow under heat and pressure in (a) above
are selected from the group consisting of polyester,
polyurethane, epoxy, polyvinyl chloride, and
acrylic, or mixtures thereof. It is understood by

the expression "melt and flow" that many liquid
materials need no further melting in order to flow
sufficiently. In order to achieve the brilliant
visual pearlescent effect, it is important that the
coating resin be a resin having a refractive index
in the finished cured laminate dissimilar from the
refractive index of the pearlescent ink on the
decorative facing sheet being used.

The binding material or suspending agent is preferably a mixture of micro-crystalline cellulose with a minor amount of carboxy methyl cellulose; "AVICEL" is sold as a mixture of approximately 89% micro-crystalline cellulose and 11% carboxy methyl cellulose. The coating composition suitably contains 1-8 parts by weight of "AVICEL" to 4-32 parts by weight of the combination of the mineral particles and polymer particulates preferably at a

ratio of mineral particles to binding material or suspending agent of 4:1 to 1:2, and a quantity of 1 part of "AVICEL" per 2 parts of mineral particles has been found to be particularly suitable. It is also possible to add small additional quantities of carboxy methyl cellulose (or none whatsoever) and a small quantity of silane as binder materials. It is preferable to include a small quantity of surfactant, as disclosed in U.S. Patent

10 No. 4,255,480, and a small quantity of solid lubricant to provide scuff resistant, as disclosed in U.S. Patent No. 4,567,087.

There are six important variables in the formulation, three of which are independent and

15 three of which are dependent. The data presented in Table 1, below, helps define the parameters. Decor paper weight, resin content and weight of the abrasion resistant composition are all independent of the formulation. The requirements for these variables are set by outside factors such as color, degree of final saturation, and abrasion resistance. Resin weight (dry) per ream is dependent on a combination of paper basis weight and desired resin content. Viscosity is dependent on the total volume of the mixture versus the content of

abrasion-resistant composition. For complete

saturation of the decor paper at the coater, the mixture viscosity should be less than 1000 centipoise for porous paper, preferably in the range of 50-100 centipoise depending on paper porosity.

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Table I
Coating Variable Comparison
For Coated/Saturated Decor Papers

10				
15		5 lb. olid		55 lb. Printed
20	Total % Add On (resin content) Volatile Content (approximate) Primary Resin (melamine) Secondary Resin (polyester Suspending Agent (Avicel)		52% 6% 75 lbs. 2 lbs. 0.7 lbs.	52% 6% 61 lbs. 2 lbs. 1.7lbs.
25	Mold Release (Infernol) Anti Foam Surfactant Catalyst (Naccure) Abrasion Resistant Mineral (Al <sub>2</sub> 0 <sub>3</sub> )	0.01 lbs. 0.04 lbs. 0.09 lbs.	0.02 lbs. 0.05 lbs. 0.11 lbs.	0.011bs. 0.04 lbs.
30	Total Coat Weight per 3000 sq. ft.	65.21 lbs.	78.08 lbs.	69.54 lbs.
35	Viscosity of formula required for good saturation Approximate viscosity prior to addition of	50-100cps	80-100cps	50-100cps
40	water Approximate water added to Reduce to 50-100 cps	400 cps 75 lbs.	300 cps 60 lbs.	1800 cps 90 lbs.

From Table I above, it will be noted that the higher the basis weight of the decor paper, a greater volume of liquid resin is required. This yields a corresponding lower final viscosity on the 80 pound paper coating as compared to the 65 pounds paper coating.

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One preferred embodiment of the present invention uses finely ground particulates of polyester resin applied at a rate about two pounds per ream of decorative laminate facing sheet. Either thermoplastic or thermoset resins may be used and the selection of which, depends on the final physical or chemical properties desired. Other embodiments include the use of polymer particulates made from polyurethane, epoxy, polyvinyl chloride, melamine and acrylic resins, or mixtures thereof in a melamine or a polyester resin. It is also possible to apply the coating resin in an amount as low as one pound per ream and as high as sixty pounds per ream of decorative laminate facing sheet.

The following examples are offered illustratively:

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### Example I

This example illustrates one method and composition

that achieves a pearlescent appearance on a laminate surface. Warm 150 gal. melamine resin at 100°F ± 5°F is placed in a container under a low shear mixer. The melamine has a density of 1.15 and 37.7% solids. TRITON CF21 surfactant in an amount of 0.001 part by weight is added per 192.8 lbs. of liquid resin. Mixing is continued at a high speed for 5 minutes. 9.86 lbs of AVICEL and 0.87 lbs Emerest 2652 (anti-foam) are rapidly added in a manner as to avoid clumping or the formation

of lumps. Immediately thereafter 38.76 lbs of polyester particulates made from the Morton 23-9036 and 24.66 lbs of 45 alumina are added rapidly and completely in less than three minutes.

The viscosity is measured and 70 gal. of water is added to provide a viscosity of no greater than 150 centipoise (Brookfield viscometer #3, spindle at 12 rpm).

Printed decor paper weighing 65 lbs/ream is coated with the composition at the rate of 196.1 lbs/ream. This gives an approximate 2 lbs/ream coating of the polyester resin. A ream of paper in the present field is 3,000 ft<sup>2</sup>. The paper is dried at an elevated temperature and is ready for use in the manufacture of laminates. The laminate was prepared in the usual practice.

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### Examples II, III, IV and V

Example I was followed above using 35.2 lbs of Glidden 2C-114 (epoxy), 4C-104 (acrylic), 5C-104

20 (polyester) and Morton Polyester 23-9036 in the following mixtures:

#### Batch Formulations

25		II	III	IV	V
	Melamine resin (liquid)				
	63% solids 15	50 gal.	150 gal.	150 gal.	150 gal.
	Water	70 gal.	70 gal.		
	Emerest 2652 Surfactant 3		3.5 lbs.		
30	Avicel 11	1.0 lbs.	11.0 lbs.		11.0 lbs.
	Aluminum oxide, 70	0.5 lbs.	70.5 lbs.	70.5 lbs.	70.5 lbs.
	40 micron				
	Mold release (Infernol)	l lbs.	l lbs.	l lbs.	1 lbs.
	Morton polyester 23-9036	35.2 lbs.			
35	Glidden polvester 5C-104		35.2 lbs	·	

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Glidden acrylic 4C-104 -- -- 35.2 lbs. -- Glidden epoxy 2C-114 -- -- 35.2 lbs.

The following table illustrates by comparison how

5 well the present invention achieves the international standards for horizontal laminate surfaces while retaining brilliant visual effects.

### Pearlescent Printed Paper

#### Typical Values

10

	Composition NEMA Test Methods	NEMA Standard	No Overlay	With Overlay	A
15	Wear value High-temp resistant Hot water	400 cycles/min. ce Slight NE*	. 25 c/m NE NE	450 c/m NE NE	NE
	Dimensional change Impact	.5 MD/.9 CD 50 in. min.		.06/.69 66 in.	.06/.69
20	Conductive heat Cigarette resistant	NE	NE 220 min.	NE	
	Stain NI	E:1-23/Mod:24-29	) NE	NE	NE
25	Scuff resistance	NE .	Severe	NE	NE
	Visual appearance		Bright- Excellent Pearles- cent	Visual	Bright- Excellemt Pearles- cent
30			Appearance	9	Appearance

\*NE = No effect
"No Overlay" is a melamine surface alone.
"With Overlay" is a standard construction of an alpha-cellulose
paper impregnated with melamine on the surface of the laminate.

This comparative test illustrates
the advantages of the present invention. The
pearlescent printed paper without a protective
overlay has a desirable appearance but lacks

required durability. The standard construction with an overlay has desirable durability but lacks the brilliant pearlescent appearance.

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It is only with the present invention, Composition A, that both the desired durability characteristics is achieved in a laminate having a brilliant pearlescent appearance.

### 5 <u>Example VI</u>

The following coating surface dispersion formula is used in the two step laminate process wherein a surface coating dispersion is applied to the exterior surface of the decorative facing sheet

10 which has been applied into the exterior side of the backing layer. After each decorative facing sheet was coated with the surface coating mixture, the coated decorative sheet was dried in the usual manner whereupon the coated decorative sheet was

15 saturated with melamine thermosetting resin and pressed to form the laminate.

### Coating Surface Batch Formulation

20	Cold Water CMC-7M AVICEL Alumina particulates,	417 2.5 7.5	grams grams grams
	20 microns	30	grams
25	Morton Polyester 23-9036	30	grams
	Ultraviolet tracer PWA @100%	0.28	grams
	Acetic Acid @5.6%	0.95	grams
	Formaldehyde @37%	0.28	grams

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	Woodgrain-2 lbs/ream)	US20 (3.5 lbs/rear	m)US40 (7.0
5	Initial Point Final Point Wear Value	125 200 163	50 275 163
10	Woodgrain-3 lb/ream)	US80* (14.3 lb/rea	ım)US90* (15.5
	Initial Point Final Point Wear Value Rate of Wear	100 500 300 0.036 grams	125 525 325 0.037 grams
15	*Mayer Bar Coating	-	,

\*Mayer Bar Coating Technique. It is understood by those skilled in the art that this is a technique to vary coating weight.

20

### EXAMPLES VII - IX

The Coating Surface Batch Formulation provided in Example VI can be prepared substituting the 30 grams of Morton Polyester 23-9036 with the polymer particulates made from the following resins:

Example VII	30 grams	Glidden Polyester 5C-104
Example VIII	30 grams	Glidden Acrylic 4C-104
Example IX	30 grams	Glidden Epoxy 2C-114

30

### EXAMPLES X - XVI

Additional coating surface mixture formulas are possible. Using the method as explained in Example I, above, the components may be mixed as follows:

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65 lb/ream paper

		Impregnating Resin	Polymer Particulate	Surfactant	Antifoam	Mineral Particulate	Diluent <sup>1</sup>
	Χ.	Polyester 61 lbs. (dry)	Epoxy 2 lbs. (dry)	0.01 lbs.	0.04 lbs.	5.0 lbs.	as required
	XI.	Polyester 61 lbs. (dry)	PVC 2 lbs. (dry)	0.01 lbs.	0.04 lbs.	5.0 lbs.	as required
5	XII.	Polyester 61 lbs. (dry)	Acrylic 2 lbs. (dry)	0.01 lbs.	0.04 lbs.	5.0 lbs.	as required
	XIII.	Acrylic 61 lbs. (dry)	Polyurethane 2 lbs. (dry)	0.01 lbs.	0.04 lbs.	5.0 lbs.	as required
	XIV.	Polyester 61 lbs. (liquid @ 100% solids)	Polyester 2 lbs. (dry)	0.01 lbs.	0.04 lbs.	5.0 lbs.	as required
	XV.	Melamine 61 lbs. (dry)	Polyester 1 lb. (dry)	0.01 fbs.	0.04 lbs.	5.0 ibs.	as required
	XVI.	Melamine 61 lbs. (dry)	Polyester 10 lbs. (dry)	0.01 lbs.	0.04 lbs.	5.0 lbs.	as required

10

#### Example XVII

Any of the resin mixtures provided in Examples I through XVI could be used in a low pressure laminate for a particle broad backing layer. A low pressure laminate would be formed using approximately 1 to 2 minute press cycles at approximately 150 to 400 psi and at a platen temperature of about 350° to 400°F. In a low pressure laminate, the polymer particulate may be a reactive resin, for example a polyester with a blocked isocyanate such as MONDUR or an

<sup>1</sup> It may also be desirable to use a suspending or binding agent such as a film forming binder microcrystalline cellulose, hydroxyethyl cellulose, carboxy methyl cellulose or polyvinyl pyroladone in quantities of from approximately 1 lb. t055 lbs. as meeded.

acrylic with a blocked isocyanate or peroxide catalyst.

### Examples XVIII - XXIII

The following coating slurries may be used in the methods illustrated in Figures 3.

		Polymer Particulate	Surfactant	Antifoam	Mineral Particulate	Diluent	Binder
	XVIII	Epoxy 2 lbs. (dry)	0.01 lbs.	0.04 lbs.	0.0 lbs.	100 lbs. water	5 lbs. CMC <sup>2</sup>
	XIX	PVC 2 lbs. (dry)	0.01 lbs.	0.04 lbs.	5.0 lbs.	100 lbs. water	5 lbs. CMC
	XX	Polyester 2 lbs. (dry)	0.01 lbs.	0.04 lbs.	5.0 lbs.	100 lbs. water	5 lbs. CMC & 2 lbs. Avicel
10	XXI	Polyurethane 2 lbs. (dry)	0.01 lbs.	0.04 lbs.	5.0 lbs.	100 lbs. toluene	6 lbs. urethane
	XXII	Polyester 45 lbs. (dry)	10 lbs.	1.0 lbs.	5.0 lbs.	100 lbs. water	5 lbs. melamine 5 lbs. HEC <sup>3</sup>
	XXIII	Acrylic 2 lbs. (dry)	0.01 lbs.	0.04 lbs.	5.0 lbs.	100 lbs. water	2 lbs. melamine resin & 5 lbs. PVP <sup>4</sup>

### Example XXIV

15

A damage resistant coated decorative facing sheet can be created by increasing the content of the substantially uncured resin in Examples XVIII through XXIII to more than 2 lbs., preferably more than 10 lbs., and most preferably to about 45 to 60 lbs. In Examples VI-IX, the quantity of the polymer particulate can be increased to 300 grams and more

<sup>&</sup>lt;sup>2</sup> CMC = carboxy methy cellulose

<sup>&</sup>lt;sup>3</sup> HEC = hydroxyethyl cellulose

<sup>25 4</sup> PVP = polyvinyl pyroladone

preferably to 600-900 grams to achieve a damage resistant coated decorative facing sheet. By increasing the weight of particulate resin used, the sheet can be flexed without resulting in damage, 5 thereby decreasing waste in production operations. A laminate can then be formed from the facing sheet without a deleterious affect in the final product. While it may be possible to achieve a damage resistant coated decorative facing sheet using any 10 method of the present invention, it is preferably achieved using the Two Step Coating and Drying Process and the Transfer Sheet Process illustrated in Figures 2 and 3, respectively.

#### 15 Example XXV

A damage resistant coated decorative paper can be created by increasing the content of the surface coating particulate resin in Examples I through XIV to a higher level and decreasing the content of the impregnating resin up to zero pounds. When the impregnating resin content is reduced and the surface coating particulate resin content increased, the polymer particulate will act as both the surface coating resin that melts and flows under heat and 25 pressure and the impregnating resin. The laminate can be prepared in the usual way.

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#### Example XXVI

When using the methods described in Figure 5 the resin coating formulas for the one step process provided in Examples II - V and Examples X - XVI can be used for coating both sides of the decorative facing sheet. Furthermore, when using the two sided coating of Figure 5, the resin coating formulas of Examples II - V and VII - XIII would be used as the top coating (61). Back coating (63) may be the same formulation without the aluminum oxide.

### Example XXVII

When using the dry coating method illustrated in

Figure 4, the particle resin can be applied at an application rate of 0.5 lb./ream up to 20 lb./ream. The particle resin that can melt and flow under heat and pressure can be selected from the group consisting essentially of polyester, melamine, acrylic, polyvinyl chloride, epoxy, polyurethane and mixtures of two or more of the foregoing.

The formulation for the impregnating resin composition that is coated on the decorative facing sheet (42) can be formulated to meet the aethestic, chemical and physical demands of the final products.

For example, the formulation provided in Examples I

- XVI, without the polymer particulate, is such a suitable formulation.

10

What is claimed is:

- 1. A coated transfer sheet for use with a decorative facing sheet to provide a decorative laminate sheet suitable for pressing, comprising:
- 5 a transfer release sheet having two exterior surfaces, and
  - a coating applied to one surface of the transfer release sheet, said coating comprising a surface coating resin that melts and flows under heat and pressure during lamination to impart one or more of the following properties: enhanced wearability, chemical, thermal, or ultra-violet radiation resistance or abrasion resistance.
- 2. A transfer sheet in accordance with claim 1, wherein the surface coating resin is a polymer selected from the group consisting essentially of polyester, polyurethane, epoxy, polyvinyl chloride, acrylic, and mixtures of two or more of the foregoing.
- A transfer sheet in accordance with claim
   wherein said coating further includes a mixture of an abrasion resistant hard mineral having a particle size of between about 1-200 microns in a
   concentration sufficient to provide abrasion resistance without interfering with visibility.

- 4. A transfer sheet in accordance with claim

  3, wherein said abrasion resistant hard mineral
  particles are selected from the group consisting
  essentially of alumina, silica, zirconium oxide,
  cerium oxide, glass beads, diamond dust and mixtures
  of two or more of the foregoing.
- 5. A decorative laminate sheet produced from the transfer sheet prepared in accordance with claim 1.
- 10 6. A method for providing a decorative laminate sheet suitable for pressing from a decorative facing sheet, said laminate sheet having a surface coating, said method comprising:
  - (a) impregnating the decorative facing sheet with an impregnating resin;
  - (b) preparing a coating surface dispersion of at least one coating resin that melts and flows under heat and pressure, suspended in a diluent with a suitable binding material, said binding material being compatible with said impregnating resin and capable of withstanding subsequent laminating conditions and said coating resin being dissimilar from said impregnating resin;
- 25 (c) coating on a first exterior surface of a laminate transfer sheet with said dispersion,

such that a surface coating layer of said coating resin is provided in an amount of from about one pound to ten pounds per ream of the transfer sheet;

- (d) drying said coating in a manner such that said coating resin is bound to the exterior surface of said transfer sheet;
- (e) placing said coated transfer sheet
  onto the resin impregnated decorative facing

  sheet with the exterior surface coated having
  the surface dispersion adjacent to the
  impregnated decorative facing sheet to obtain a
  decorative laminate sheet suitable for
  pressing.
- 7. The method according to claim 6, wherein the step of preparing a coating surface dispersion further comprises selecting a resin that melts and flows under heat and pressure selected from the group consisting essentially of polyester,
- 20 polyurethane, epoxy, polyvinyl chloride, acrylic and mixtures of two or more of the foregoing.
  - 8. The method according to claim 7, wherein said coating dispersion further includes a mixture of an abrasion resistant hard mineral having a particle size of between 1-200 microns in

concentrations sufficient to provide abrasion resistance without interfering with visibility.

- 9. The method according to claim 8, wherein said abrasion resistant mineral particles are selected from the group consisting essentially of alumina, silica, zirconium oxide, glass beads, diamond dust and mixtures of two or more of the foregoing.
- 10. The method according to claim 9, wherein said impregnating resin is melamine and said abrasion resistant mineral is alumina which is chemically bound to said melamine with a silane.
  - 11. A laminate produced from the decorative laminate sheet prepared in accordance with claim 6, wherein the decorative laminate sheet is laminated to a backing layer under heat and pressure.
  - 12. A method for providing a decorative laminate sheet suitable for pressing from a decorative facing sheet, said laminate sheet having a surface coating, said method comprising:
    - (a) coating a decorative facing sheet with a liquid impregnating resin coating mixture; and
- (b) depositing surface coating resin25 particulates that melt and flow under heat and

15

pressure onto the coated decorative facing sheet; and

- (c) drying the coated decorative facing sheet having particulate deposits to obtain a decorative sheet suitable for pressing.
- 13. The method according to claim 12, wherein the step of depositing surface coating resin particulates onto the decorative facing sheet further comprises selecting the particulate resin from the group of polymers consisting essentially of polyester, polyurethane, epoxy, polyvinyl chloride acrylic and mixtures of two or more of the foregoing.
- 14. A laminate produced from the decorative

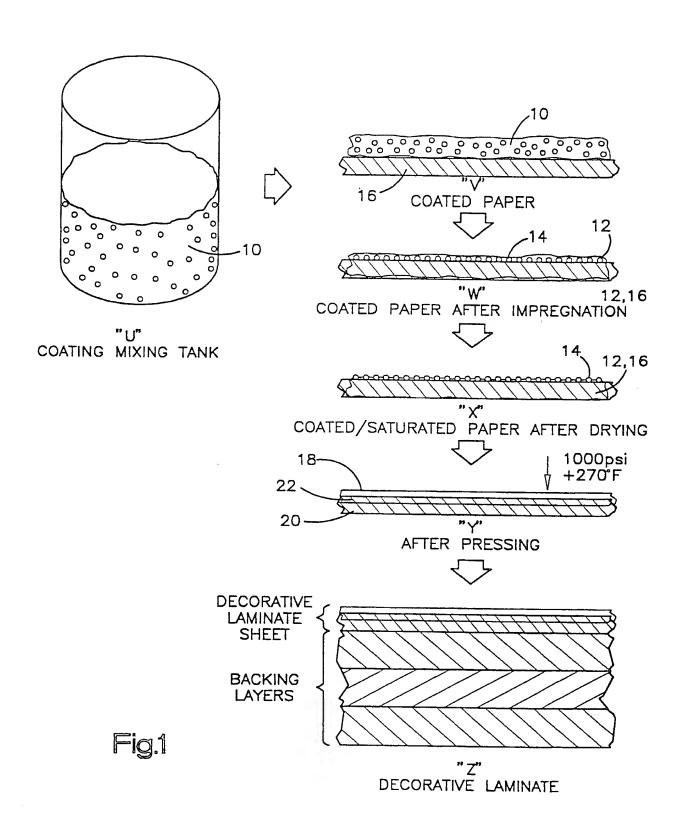
  15 laminate sheet prepared in accordance with claim 12,

  wherein the decorative laminate sheet is laminated

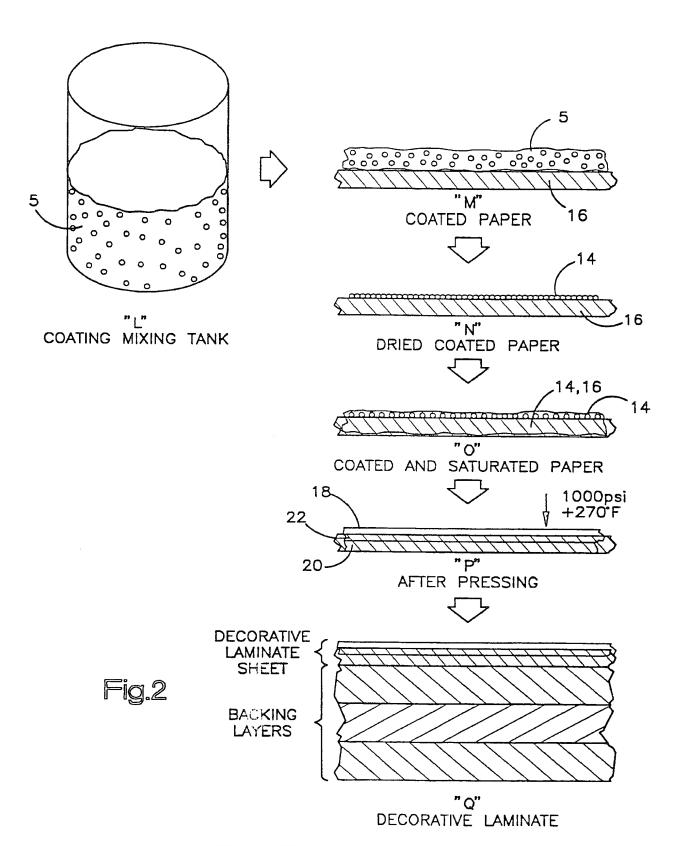
  to a backing layer under heat and pressure.
  - 15. A method for providing a decorative laminate sheet suitable for pressing from a decorative facing sheet, said method comprising:
    - (a) providing a decorative facing sheet having a top external surface and a bottom external surface;
- (b) applying a first slurry mixture
  comprising surface coating resin particulates
  to one of the surfaces;

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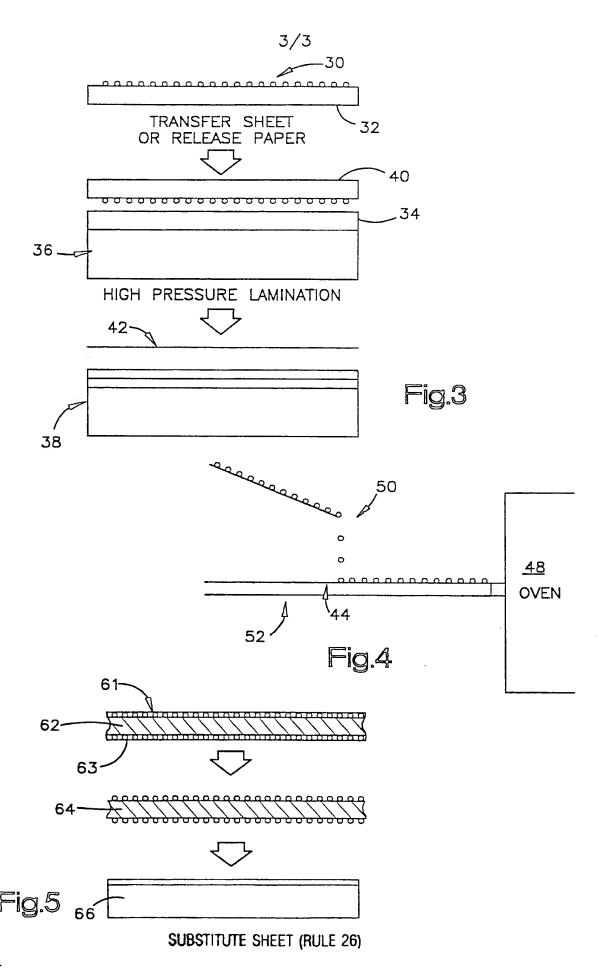
- (c) applying a second slurry mixture comprising an impregnating resin to the other surface;
- (d) drying the coated decorative sheet having particulate deposits to obtain a decorative sheet suitable for pressing.
- 16. The method of claim 15 wherein the first and second slurries have different compositions.
- 17. The method of 16 wherein the first and 10 second slurries have the same composition.
  - 18. The method of claim 16 wherein the first slurry comprises melamine.
- 19. The method of claim 14 wherein the first slurry comprises at least two dissimilar resins, one of said resins being a liquid melamine impregnating resin and the other being a surface coating resin that melts and flows under heat and pressure during lamination to form a laminating surface having one or more of the following properties: enhanced versatility, chemical, thermal, ultra-violet radiation resistance or abrasion resistance.
- 20. A laminate produced from the decorative laminate sheet prepared in accordance with claim 14, wherein the decorative laminate sheet is laminated to a backing layer under heat and pressure.



SUBSTITUTE SHEET (RULE 26)



SUBSTITUTE SHEET (RULE 26)



A. CLASSIFICATION OF SUBJECT MATTER IPC 6 B44C5/04 D21H27/26

C. DOCUMENTS CONSIDERED TO BE RELEVANT

B32B27/08

According to International Patent Classification (IPC) or to both national classification and IPC

#### **B. FIELDS SEARCHED**

Minimum documentation searched (classification system followed by classification symbols) IPC 6 B44C D21H

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	US,A,4 765 858 (H. F. E. VANKERCKHOVEN ET AL) 23 August 1988	1,5,6,11
	see column 2, line 21 - column 5, line 63	
X	US,A,4 376 812 (W. W. WEST) 15 March 1983	1,5,6, 11,12, 14,20
	see column 3, line 6 - column 7, line 66; example 1	14,20
X	EP,A,O 189 070 (TECHNOGRAPHICS FITCHBURG COATED PRODUCTS INC.) 30 July 1986 see page 8, paragraph 3 - page 13,	1-5
A	paragraph 1; example 1	6-9,11
	_/	

Further documents are listed in the continuation of box C.	Patent family members are listed in annex.
*Special categories of cited documents:  'A' document defining the general state of the art which is not considered to be of particular relevance  'E' earlier document but published on or after the international filing date  'L' document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)  'O' document referring to an oral disclosure, use, exhibition or other means  'P' document published prior to the international filing date but later than the priority date claimed	To later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention  "X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone  "Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.  "&" document member of the same patent family
Date of the actual completion of the international search  20 December 1994	Date of mailing of the international search report  3 1. 01. 95
Name and mailing address of the ISA  European Patent Office, P.B. 5818 Patentiaan 2  NL - 2280 HV Rijswijk  Tel. (+31-70) 340-2040, Tx. 31 651 epo nl,  Fax (+31-70) 340-3016	Authorized officer  Doolan, G

Form PCT/ISA/210 (second sheet) (July 1992)

Interr 1st Application No

PCT/US 94/09956

	ion) DOCUMENTS CONSIDERED TO BE RELEVANT	
Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
	WO,A,93 01935 (NEVAMAR CORPORATION) 4 February 1993 see page 5, line 29 - page 13, line 12; examples 1-13	12-16, 19,20
	US,A,4 726 986 (D. L. CANNADY ET AL) 23 February 1988 see column 1, line 50 - column 7, line 60; example 1	12-14, 19,20